

ORGANIC CHEMISTRY LABORATORY II

(CHEMISTRY 0124)

TENTATIVE SYLLABUS
Main Campus

Summer '06
TEMPLE UNIVERSITY

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COURSE DESCRIPTION:

This course is a continuation of Chemistry 123 in which you will utilize microscale laboratory techniques in organic chemistry introduced there. This course includes the preparation, purification, and analysis including multi-step sequences, of typical moderately complicated organic compounds. Offered year round, it places an emphasis on each student's independent skills in synthesizing and characterizing organic compounds. It meets for one session of three hours and twenty minutes twice each week during the First and Second sessions. The "general guidelines" from the first semester course still apply and may be consulted at www.temple.edu/chem-help or www.blackboard.temple.edu

Co requisite: Chemistry 122 - Organic Chemistry Lecture II (Note: This course requires successful completion of both Organic Chemistry 121 and Chemistry 123.) The minimum grade requirement is C- or better in BOTH courses. Failure to meet these standards is grounds for dismissal from this course.

TEXTBOOK and SUPPLEMENTAL MATERIALS:

- 1). Mayo, D W, *et al.* "**Microscale Organic Laboratory . . .**", 4th edition, J. Wiley & Sons, New York, 2000.
- 2). Eye Protection that meets ANSI standards (and other Personal Protective Equipment as you desire).
- 3) "**General Safety Guidelines for CST Labs**" including the release waiver**
- 4). A hardbound composition book to serve as permanent lab notebook.

SCHEDULING:

The **FIRST LABORATORY DAY IS WEDNESDAY, 5 July 2005.**

Your Organic Chemistry 124 lab is scheduled for two meetings per week. Students are expected to arrive promptly with a carbon copy of the pre-lab write up appearing in their lab notebook. You should also be prepared for the quiz that begins every lab. By the scheduled end of class, students will have cleaned their bench spaces and returned equipment as necessary. Your timely attention to these responsibilities will be rewarded.

There is no provision for a make up lab or to "make up" of a missed quiz. As a result of an absence you will have lost the opportunity to accumulate points towards your final total and grade. *When you return from an absence, come prepared to undertake the laboratory work scheduled for the time noted...not for what you missed!*

Your daily schedule is also presented on the web. If you are using www.blackboard.temple.edu, you may access your account to find that are scheduled plus comments on many of the labs. For those not using Blackboard, you are reminded of the Department's help page - www.temple.edu/chem-help

As in Chemistry 123 this course will include the opportunity to utilize the Educational Technology Center, Ground Floor Anderson Hall. In this course we will use a software self pace learning package, "Spectroscopy" in addition to the Alchemy III program you learned last semester. **YOU WILL BE AT A DISTINCT DISADVANTAGE IN THE LECTURE PORTION OF THE COURSE WITHOUT THIS MATERIAL!**

Most of the term will be spent in the laboratory to which you are now assigned doing BENCH WORK - more traditional for this course. You should plan now on having your "Safety Guidelines for CST Labs" and EYE PROTECTION (SAFETY GLASSES OR GOGGLES) READY; YOU WILL NOT BE PERMITTED GLASSES READY IN THE LABORATORY UNLESS YOU ARE WEARING EYE PROTECTION!

Laboratory Schedule: [There is a new Lab each DAY.]

Before you come to class, you must read and sign the appropriate safety acknowledgment to be found in the "GENERAL GUIDELINES FOR CST LABORATORIES" - available in CONWELL 601. This RED SHEET will be collected during lab check-in and is required to continue in this course. You will not be permitted to work beyond May 21 without a current copy on file.

LAB MEETING

Experiment

5 July Check-in and Discussion of regarding lab policies, notebooks, grading and safety. Review stoichiometry and first semester techniques (ADP use, recrystallization, etc.). Understand expectations of the Dep't, AF, ... Begin Diels - Alder Reaction of α - **Phellandrene** and **maleic anhydride** attached to this syllabus

7 July Experiment 28: Electrophilic Aromatic Substitution: Bromination of Aniline to yield 2,4,6-Tribromoaniline. See page attached or "photon.chem.temple.edu" What is the best way to characterize this product? Also collect and recrystallize your Diels-Alder product. Determine product's melting point when dry. Complete review of stoichiometry and importance of knowing physical properties. Check Diels-Alder Reaction .

12 July Experiment 16: Grignard Reaction: Preparation of Diphenylcarbinol. Do not wash your equipment. Oven dry the equipment. See attached or "photon.chem.temple.edu" WARNING: ALL FLAMES MUST BE EXTINGUISHED BEFORE ETHER IS USED. Monday, July 18, last day to drop.

14 July Experiment 22: Williamson's ether synthesis: n-Propyl β -Naphthyl Ether. Substitute n-propyl bromide for n-propyl iodide. See attached instructions or "photon.chem.temple.edu"

19 July Experiment 5B: Reduction of Ketone using a Metal Hydride Reagent: *cis*- and *trans*-4-*tert*-Butylcyclohexanol. p 139. Run IR. What other method(s) could be used to characterize your product?

21 July Experiment 32: Hypochlorite Oxidation: Cyclohexanone. p 353. Run IR

26 July Preparation of Piperonylonitriles from Piperonal, to be distributed in lab; as adapted from J Chem Ed (6). Consult www.blackboard.temple.edu [July 31: Last day to withdraw]

28 July Experiments 23B: Amide Synthesis : N,N'-Diacetyl-1,4-phenylenediamine. p 30

2 August Experiments 24A and 24B: Imide Synthesis : N-Phenylmaleimide. p. 309

4 August Experiment 20: Aldol Condensation: 4,4'-Dichlorodibenzalacetone., p 279. Recrystallize your product, then run the IR. Catch-up/Make-up sign-up. . Substitute 4-chlorobenzaldehyde for benzaldehyde. Hand-out for laboratory practical.

9 August Laboratory Practical Examination.

11 August **CHECK OUT. LAST LABORATORY QUIZ. Your notebook is due.**

GRADING:

You will be judged on your skills. Thus, the work in this laboratory is done on your own.

Course grading is as follows:

Laboratory quizzes*	30%
Laboratory Notebooks.(due at Last Quiz)	30%
Laboratory Last Quiz (written)*....	20%
Laboratory Final (practical) and technique**	<u>20%</u>
	100%

**Performance on the last quiz above minimum standards is required to pass the course.

>>*EACH LABORATORY SESSION BEGINS WITH A QUIZ*<<

***There is no make-up for missed quizzes.**

The requirements for your LABORATORY NOTEBOOK are noted below in the GENERAL INFORMATION SECTION. This notebook is to accompany you to lab every meeting and is due no later than the day of the last quiz.

**Technique will include items such as being careful to avoid contamination of common reagents, remembering to keep your work area clean, taking care of the equipment including re-hanging automatic delivery pipets, finishing on time, returning equipment to the location found, keeping only originally inventoried drawer items, *recapping reagent bottles, etc.* *The ability of a section to maintain the cleanliness around balances, sorting paper*

Grading:

continued:

waste, sharps, and broken glass and then placing these items into the proper container will provide a measure for that section's average.

Final totals are checked for individuals who are near grading boundaries and may have missed a quiz.

INCOMPLETES / WITHDRAWALS:

Please note that an Incomplete (I) is only to be given in accord with institutional procedures and which is not fulfilled until the specific requirements have been met and forms signed and submitted. The Temple University Policy on "Incompletes" governs this course (http://policies.temple.edu/getdoc.asp?policy_no=03.12.13). Additionally, the grade of incomplete, I, will be considered only in those cases where at least 40% of the term's work has already been completed, and where there is a valid excuse (medical or similar) for missing the remainder of the course. The fear of earning a poor grade is not considered a valid excuse. For those students who are assigned a grade of "I", all previous scores will stand and be used in the calculation of the final score when the course is completed. Students wishing to pursue an incomplete must obtain an Instructor Approval for an Incomplete Form (available from the web page) that the student and his or her instructor(s) must complete, before taking it to Dr. Findeisen to be used to draft the official incomplete contract. Only Dr. Findeisen can sign and process incomplete contracts.

Please note that a withdrawal (W) is an institutional procedure that is not complete until the withdrawal form has been signed and submitted to the Registrar's office. Temple University Policy on "Withdrawal" found at http://policies.temple.edu/getdoc.asp?policy_no=02.10.14 governs this course.

EXAMINATION POLICY:

Each meeting there is a lab quiz. The cumulative quiz total represents 30% of a student's final grade. There are no make up quizzes. If a quiz is given prior to your arrival, it is considered missed. Instructors are asked to wait five minutes before administering the quiz. In general, a single missed quiz should not alter a grade. An estimate of the potential score can be obtained by comparing the student's individual rank within the section to that section's performance on the specific test. Students are advised to keep all quizzes to aid in preparation for the last quiz.

This last laboratory quiz occurs on the last scheduled meeting of the student's section. It is usually a quiz of 10 or more questions relating to the experiments performed during the semester. One or more questions may include stoichiometry, IR and NMR spectroscopy. A minimum grade on the final is expected for those who will receive grades higher than a B⁺.

SAFETY REQUIREMENTS:

Although the Department is sensitive to the need for demonstrating personal freedom, the laboratory can be a dangerous place for its expression. Therefore, in addition to denying you admission should you refuse to wear eye protection, the Department requires appropriate attire, specifically

1. long hair be tied back
2. closed footwear be worn (open-toed shoes/sandals are not acceptable)
3. scarves, veils, etc. be tied back or removed during the lab.

ATTENDANCE POLICY:

Simply stated, you must attend class to perform the experiments. You will be asked to leave the class if your pre-lab preparation is insufficient, if you do not dress appropriately or lack eye-protection, or arrive when there is insufficient time to perform the experiment. Missing more than one quiz and failing to write up a lab may effect your grade. One reason is that the lab you have missed will be included on the last lab quiz.

When you return from an absence, come prepared to undertake the laboratory work scheduled for the time noted...not for what you missed! IF YOU ANTICIPATE BEING THIRTY (30) MINUTES OR MORE LATE TO THE LABORATORY, DO NOT BOTHER COMING. YOU WILL NOT HAVE TIME TO DO THE EXPERIMENT!

COURSE GOALS:

You will be learning experimental organic chemistry at the microscale level. This means you will be working with very small amounts of materials and may become able to observe and to learn more organic chemistry in a two semesters than many of previous students learned in nearly two years. Hopefully you will find this laboratory an exciting, interesting and surprisingly pleasant adventure.

The course is structured to assist you develop skills in several areas considered in lecture and the lab.

1. Molecular modeling programs will be introduced and available to enable you to construct and manipulate structures considered in lecture or your lab texts.

Course goals

Continued:

2. General safety protocols for the laboratory will be enforced.
3. A formal, permanent, hardbound laboratory notebook will be maintained with a detailed pre-lab copy prepared in order for each student to perform successfully in the weekly quiz on the experiment to be performed.
4. Techniques and microscale organic lab skills will be developed to permit the flexibility of choosing your own scaling sequence without being tied to a prescribed set of quantities.
5. Methods of characterization of organic materials at the microscale will be utilized.
6. Successful completion will provide a foundation from which you can develop an expertise in microscale techniques as well as the confidence gained by mastering any challenging program.

This course is designed to allow the interested participant to develop rapidly the skills needed to slice more deeply into organic chemistry than ever before. Attendant benefits are greater confidence and independence in acquired laboratory techniques.

GENERAL INFORMATION FOR THE CHEMICAL LABORATORY

Recognizing its obligation to your safety and the environment, and noting the general reduction in the scale on which organic reactions are run in industrial research laboratories that has accompanied the revolution in analytical procedures, the Department of Chemistry has obtained funding from the University to convert your Organic Chemistry Laboratory from one that uses relatively large quantities of material and large (or macro scale) equipment to one that uses small quantities of material and small (or microscale) equipment. In the microscale laboratory reduction in the quantities of materials used is dramatic and, generally, the time required to carry out reactions is also reduced. Your manipulative skills will be tried.

Our earlier experiences with this course have taught us that we must tell you that it is *critical* that you read, outline, and understand the manipulations you are to perform before you come to class. Processes on a small scale occur with rapidity. There is no time to study the book while reactions are taking place.

There is **NO** catch-up/make-up laboratory scheduled. If you are absent from your laboratory class, be prepared to perform the current lab on the syllabus for the day you return not the missed work. You will have missed a lab, its quiz, and the opportunity to score the associated points.

YOUR NOTEBOOK:

The notebook will be brought to every lab so that it can be kept current. It may be collected or reviewed at any time. A carbon copy of the pre-lab write up will be submitted in the event your original is misplaced or lost. The format and list of ten essentials for each notebook write-up are found within the lab text, Mayo et al., 4th ed. Maximum points are awarded only when notebooks are completed and submitted in a timely manner.

To help you understand the need to know what you are going to do before you begin...

a) You must write-up (in **INK AND IN YOUR HARDBOUND (NOT SPIRAL) NOTEBOOK WHILE MAKING A CARBON COPY TO BE TURNED IN**) what you anticipate doing in the laboratory before class time. A typical write-up will be found in your text [p. 30 - 31]. Clearly, you will not be able to write-up the results, but you can, and should, indicate quantities of materials to be used, what the reaction or procedure is, and how the equipment set-up will appear. [First six "Key component of Lab Experiment write up items", p. 29.]

b) **The carbon copy of the preliminary write-up will be examined by your instructor before you start the experiment. If you do not have that material when you come to lab you will not be permitted to begin the experiment!** Laboratory notebooks will be collected and graded at least twice during the term. Your instructor will work out a schedule with you.

THE QUIZZES:

At the beginning of every laboratory period, while your teaching assistant is looking through the carbon copy of the pre lab write-up, you will be taking a short (ca. 10 min) **QUIZ** dealing with the manipulations you have prepared to do. The quiz will be given about five (5) minutes after the laboratory period is scheduled to start. If you are late, you will miss the quiz. **There are no make-ups**. The sum of all quizzes will account for 30% of your grade.

GRADING FOR THE PRACTICUM:

Determination of grades for the laboratory. practicum:	
Answers to assigned questions	25%
Calculations	10%
Sketch of the Apparatus	10%
Product	25%
Lab technique/safety/questions by instructor	<u>30%</u>
TOTAL	100%

ASSISTANCE:

The Math and Sciences Resource Center will be open to support Organic Chemistry this semester. Its URL is www.temple.edu/msrc. It is strongly suggested you determine what *free services* the MSRC providing this course. Traditionally study groups and one - on - one tutoring have been established.

DISABILITY RESOURCES and SERVICES:

Located in 100 Ritter Annex, this Office of Empowerment arranges accommodations and provides information and support in accessing University programs, facilities, and activities for students with 'certified' disabilities. Services include assisting with academic adjustments and accommodations including sign language interpreters, test proctoring, library research, note taking, and reader services. Information on mobility, wheel chair storage, adaptive computing, small equipment loan, specialized scholarship, and career/internship resources is also available. URL - www.temple.edu/disability ; 215.204.1280; TTY at 204.1786; FAX at 204.6794 Students intending to utilize these services should introduce themselves to Dr. F. early in the semester in order to utilize the fullest capabilities of these resources.

Other Observations and Comments:

On behalf of the faculty of the Department, Drs. Dalton, Davis, Krow, Sieburth, Williams, Das, Przeslawski, and Hill have agreed to participate in this laboratory experience with you and, on any given day, you may expect to find at least one of them in your laboratory sometime during the period.

This syllabus was prepared 20 February 2005 and before the determination of Chemistry 124 instructors. As a result all information is **tentative** and it will change. A new experiment, now in development stage, may be adopted on this date. Visit your "**Blackboard**"™ site or **/chem-help** for more recent announcements.

Our experiences have found well prepared students to be more successful in this course than in the co requisite lecture. For this reason a commitment agreement has been attached to this syllabus whereby each student earns credit for acknowledging the expectations in lab and lecture courses in a timely manner. Return your completed form to Dr. F. this week. Typically a quarter to one third of the students beginning Organic Chemistry each semester do not receive the grade of C- or higher. It is our desire to increase the success rate by alerting all students to our expectations.

Questions concerning this syllabus may be addressed to Dr. Findeisen (afindeis@temple.edu) at 215-204-7161 in Beury Hall 400. During the first three meetings of each summer semester, Dr. F. conducts Alchemy III (Molecular Modeling) instruction when Chemistry 123 sections meet. He can often be located in fourth floor teaching labs within Beury Hall if you can not find him in his office. If you wish to find him for a "Green Card" for a closed section, it is best to arrive at the section you are seeking, so "noses" can be counted and compared to the class list. Your continued patience and cooperation are appreciated.

To Summarize:

This laboratory course, the companion course to CHEM 122: Organic Chemistry Lecture II, introduces the practice of organic chemistry in the laboratory. In this second semester course the primary emphasis is on applying basic laboratory techniques such as extraction, recrystallization, steam distillation, reflux, gas to synthetic chromatography as well as FT-IR spectroscopy. Students will use software to learn molecular model building fundamentals. An attempt is made to correlate the syllabus topics with those being considered simultaneously in lecture.

Course syllabi do not constitute a contract with students and do not supercede University policies.

PREPARATION OF 2,4,6-TRIBROMOANILINE

Initiate the reaction in the hood. In a 5-mL conical vial fitted with a cap and spin vane, place 100 μL of aniline. Use a calibrated plastic pipet to add 0.8 mLs of brominating solution to the reaction vial in the hood. Cap the reaction vial and magnetically stir the mixture for five minutes. When uncapped, HBr will be released. If the spin vane cannot stir the mixture open the vessel under the hood from time to time and mix it carefully with a Pasteur pipet.

Add 2 mLs of water to the reaction mixture using a calibrated pipet. If the solution is highly colored, you may use a little 30% sodium bisulfite to discharge the color. Collect the solid product by suction filtration in a Hirsch funnel and wash it twice, using a few drops of cold 50% ethyl alcohol each time.

Recrystallize the crude product from 1.5 – 2.5 mLs of 95% ethyl alcohol in a Craig tube. A test tube and Hirsch funnel can also be used. Dry and weigh the product.

Analyze the product. Determine the melting range and calculate the percent yield assuming aniline is the limiting reagent.

SUMMARY

1. (Hood) Place one reagent in the reaction vessel and add the brominating reagent with stirring.
2. After standing with stirring add water.
3. Decolorize if necessary
4. Collect and wash the crude product.
5. Recrystallize from 95% ethanol.
6. Analyze the product

QUESTIONS

1. What type of director is the $-\text{NH}_2$ group?
2. Why could sulfuric acid not be used in place of acetic acid?
3. The color in the reaction may be due to the oxidation products of aniline or excess bromine? What is the function of the bisulfite? What does it become?
4. How are insoluble impurities removed?
5. How could p-bromoaniline be prepared?

PREPARATION OF DIPHENYLCARBINOL (GRIGNARD REACTION)

"The Grignard reagent, as generally understood, is an organomagnesium halide-ether complex that is formed by the action of magnesium with an organic halide in a solvent such as ether. It can react with some elements and a variety of compounds to yield (when not involving simple metathesis) reaction intermediates or complexes. These complexes or the reagent itself can be hydrolyzed with dilute acids or saturated ammonium chloride solution to yield compounds in which the magnesium has been replaced by hydrogen. Compounds containing the carbonyl grouping such as aldehydes, ketones, esters, and acyl halides, are most widely used with the Grignard reagent.

1. Why not carboxylic acids?

These reactions followed by hydrolysis find synthetic use in many preparations, particularly of alcohols.

The restrictions on the choice of alkyl or aryl halides used with the Grignard reagent are that the carbon-to-hydrogen bond must not be too strong for the magnesium to break and must not be too weak lest a coupling reaction predominate. The reactivity of the halides increase in the order: chloride < bromide < iodide. The aryl chlorides, for example are not usually affected by magnesium in ether. Since the yield are said to be inversely related to the reactivities, the bromide is often the halide of choice. See J.Chem. Educ., 1989, 66, 586. All equipment and all reagents used in this preparation must be scrupulously dry.

2. Why must everything be dry?

PROCEDURE:

Assemble the apparatus and activate the magnesium. Place 50 mg of bright magnesium filings or turnings (in that or) in your 10 mL round bottom flask equipped for addition and reflux as well as a drying tube on the Claisen Head side arm.

Initiate the formation of the Grignard reagent. Add about 300 μL of anhydrous ether to the contents of the 10 mL flask. Mix 200 μL of anhydrous bromobenzene and 600 μL s of anhydrous ethyl ether in a small, dry flask and place the mixture in a calibrated Pasteur pipet. With the water flowing in the condenser, add about 100 μL s of the ether-halide mixture to the magnesium in the flask. If everything is dry and free of contamination, the ether mixture may start to boil from the heat of reaction. Discolored spots, which may appear on the metal, are among the signs of reaction. BE prepared to cool the reaction if it is too vigorous, or heat it gently if it is reluctant to start. [You can do this with the palm of your hand or by adding "kicker" provided by your instructor.]

When the reaction becomes sluggish, swirl or gently shake the mixture to ensure complete mixing. Briefly reflux the reaction mixture. When there appears to be no more reaction and much or most of the magnesium has been consumed, cool the flask in a cold water bath.

React the Grignard reagent with the aldehyde Add 190 μL of benzaldehyde dropwise through the reflux condenser with frequent shaking or swirling to mix the reagents. When the addition is complete, allow the mixture to warm to room temperature, and, with a clean dry Pasteur pipet, stir or probe the semisolid mixture to expose portions of the mixture that, because of viscosity, have not mixed and reacted. Quickly reassemble.

Break up the complex. Pour 1 mL of 3 M sulfuric acid on an ice cube to thoroughly cool it. Cautiously add the cold acid solution to the reaction mixture through the reflux condenser. Shake, swirl, and mix. If necessary, probe the mass with the Pasteur pipet to keep the reagents in contact with each other. If the upper layer is not fluid or all of the magnesium has not dissolved, add a bit more 3M acid to achieve this. This acid need not be cooled.

Isolate the product. Transfer the liquid contents of the flask to your 25-mL Pear shaped flask. Rinse the round bottom flask with a little solvent grade ether, add this to the 25-mL pear shaped flask. All of the product should be dissolved in the ether. Repeat this procedure with a little water. Separate the aqueous layer.

3. Which is the aqueous layer? How could you test it?

Wash the ether layer with an equal volume of water and retain the ether layer. Condense the ether layer. Cool and add ice to solidify the crude product. Decant the water or filter. Recrystallize the product from heptane or another hydrocarbon solvent of similar boiling point. Determine yield and melting point after washing the crystals once with a little cold solvent and permitting them to air-dry..

SUMMARY OF GRIGNARD REACTION

1. Activate the magnesium after assembling the dry apparatus. Use kicker if necessary.
2. Cool, add ether, and then add a bit of the mixture (ether and bromobenzene).
3. Continue the addition to just sustain the reaction.
4. Reflux briefly and cool.
5. Add benzaldehyde and mix thoroughly.
6. Acidify the mixture, destroying any excess metal
7. Separate the organic layer and wash it with water.
8. Evaporate the ether in the hood
[Steam distillation of impurities if any.]
9. Crystallize, and collect by decanting through a filter.
10. Recrystallize heptane.
11. Analyze the product as directed.

Question 1 through 3 in text plus.

4. Explain the method for removing the ether from the product.
5. Explain why more H_2SO_4 is added to if some magnesium remains. What is the reaction?
6. How could *p*-chlorobenzoic acid be produced by Grignard reaction from *p*-chlorobenzene? (Equation and explanation).
7. Analyze the infrared spectrum of the product. Contrast this spectrum to that expected for the starting materials.

PREPARATION OF n-PROPYL β -NAPHTHYL ETHER (WILLIAMSON ETHER SYNTHESIS)

Introduce the reagents and initiate the reaction. In a 5-mL conical vial equipped with a reflux condenser and a spin vane, place, in the following order and with mixing, 1 mL of methanol, 400 mg (2.7mmol) of b-naphthol, 400 mL (2.5 mmol) of 25% NaOH solution and 250mL (4.0 mmol) of n-propyl bromide. Use an automatic delivery pipet to dispense the liquids. Stir and reflux the mixture for 25-30 minutes in a sand bath or aluminum block.

Isolate and purify the product. Using a Pasteur pipet, quickly transfer the solution into a 5-mL conical vial (test tube, plastic centrifuge tube or small flask) containing 2 mLs of cold water and mix well. Using a pipet or plugged pipet, remove as much of the water as possible from the solid, add an additional 500 mL of cold water and transfer the wet solid and slurry to the bottom of a 2-mL Craig tube. Insert the plug and centrifuge in the usual manner. Alternatively, bend a piece of wire to create a loop and a handles. Put the loop of wire around the bottom of the Craig tube: place it into a centrifuge tube and centrifuge it right side up for two minutes. Make sure that the handle extends just out of the centrifuge tube. Remove the water by decanting it off or by using a pipet. Add about 1.7 – 1.8 mLs of 95% ethanol to the solid in the Craig tube. Boil the solution gently (stir with a microspatula) until all of the solid is dissolved. It may be necessary to add small amounts of hot solvent to dissolve the crystals. Allow the solution to cool.

Collect the crystals. If the crystals do not form by the time the tube is cool enough to hold, the solution may be “seeded” by evaporating a little of it on a spatula and by using this spatula, covered with “seed” crystals, to stir the solution. Cool the solution, and allow the crystals to form: then plug, invert, and centrifuge the Craig tube in the usual manner to collect the crystals.

Analyze the product. Allow the crystals to dry, weigh them, determine the melting range, and calculate the percent yield.

SUMMARY

1. Equip a vial for reflux and add the reactants in the order given.
2. Reflux for 20 – 30 minutes, and pipet the reaction mixture into water.
3. Remove the water from the solid and wash it again with water
4. Recrystallize from a minimum of hot 95% ethanol
5. Dry, weigh, and analyze the product.

QUESTIONS

1. Sodium hydroxide reacts with b-naphthol to form the sodium salt. Can sodium ethoxide, the sodium salt of ethanol, be formed in the same manner? If not, how is it made?
2. Explain the acidic nature of b-naphthol.
3. Why does the ether precipitate when the alcoholic solution is poured into water?
4. What else could have been used in place of n-propyl bromide to produce the same ether?
5. Outline the SN₂ mechanism for this reaction.
6. Why is sodium hydroxide used in the smallest molar amount?
7. Explain the “seeding” procedure.